BERYLLIUM FOILS FOR WINDOWS IN COUNTER OF NUCLEAR RADIATION

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Based on the optimization of the main structural characteristics (grain structure, texture, dislocation substructure) are defined modes of deformation and heat treatment of beryllium foils (purity > 99.95%), providing their excellent mechanical properties and optimized modes of deformation and heat treatment. Analyzed various technological methods rolling foils to their rational use for the practical implementation of the results of the study. It is shown that the strength and plastic properties of the foils beryllium higher than that of similar foils foreign manufacture.

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INTRODUCTION

Low coefficient of linear absorption, combined with unique physical and mechanical properties of beryllium make it perfect material for making windows in the X-ray machines and counters nuclear radiation, when used as a source of neutrons in high energy physics, semiconductor detectors, micro-contacts in radio electronics, screens in laser technology and many other critical applications.

Development of methodology for the production and study of the structure and properties of beryllium foil in KIPT has more than half a century history. At the beginning of the 60s G.F. Tihinskim with co-workers first received large quantities of beryllium foils with thickness 100 microns and a total area of several square meters by vacuum evaporation and condensation on the molybdenum substrate [1]. Then it was discovered that this method is appropriate for relatively small amounts of foils necessary for research purposes and that the foils have a lower density and a large number of pores. Although this method can get a beryllium foil with a thickness of a fraction of a micron [2], vacuum tight are only at thicknesses greater than 30…50 microns. Furthermore, the structure of foils changes with the thickness, and particularly ductility properties substantially depend on the substrate temperature remains at a relatively low level. This method does not permit a sufficiently large foils of the same thickness over the entire area and is substantially laboratory method.

Therefore, since the second half of the 60s. the last century in KIPT began extensive research on methods to rolling beryllium foils, as well as a systematic study of the influence of purity of the metal, deformation and heat treatment conditions, and other factors on the structure and properties at first thin sheet of beryllium [3], and then the thickness of thin foils of 10 microns.

In carrying out the work studied the effect of deformation conditions (temperature, degree, rolling direction) and heat treatment conditions on the structure, substructure and mechanical properties of rolled products (30…500 microns) from ingots beryllium and beryllium foil thickness influence on the mechanical properties and vacuum density.

Beryllium thin foils manufactured in the form of disks with dimensions: thickness 30…40 microns and diameter 20…25 mm.

The work was first studied the formation of substructure and texture by rolling ingots of beryllium with high degrees of deformation. Typically, the process of getting beryllium foil start with rolling beryllium sheet to a thickness of 0.5…1.5 mm, which are then repeatedly recovered in the steel covers, chemically pickled and rolled to the desired thickness. Steel covers used to prevent oxidation of beryllium and for maintaining the desired temperature, usually ranges from 400 to 850°C. It was revealed that the rolling work pieces of beryllium in covers made of stainless steel metal is saturated with chrome and foils strongly embrittle. Furthermore, serious difficulty is the separation laminated foils from the cover associated with grasping beryllium by cover material, resulting in the formation of surface and subsurface defects in the resulting foil. To solve this problem, many researchers have attempted to use various protective coating (lubricants) of zirconium, ammonium phosphate, clays, etc. However, the protective coating result in the formation of various films on the surface of beryllium which are stubborn and contribute to receiving no uniform foil thickness due to the uneven spreading of the lubricant over the surface of beryllium in the rolling process.

By U.S. patent authors [4] was achieved good separation beryllium foil from the metal shell by heat treatment, which consisted in that the laminate pack is heated to a temperature of 600…800°C and then quenched to room temperature. The specified heat treatment combined with annealing foils for its plastification.

Attempts to use as covers of material softer than stainless steel, for example, mild steel, were unsuccessful due to the strong adhesion with beryllium. Mild Steel contributed to the formation of a thicker oxide film on a beryllium foil. In addition, a "soft" cover material did not provide preserving the original shape foil rolling and led to poor quality of the surface of beryllium. However, despite these shortcomings, the rolling beryllium foil is most often used covers of mild steel.

Although beryllium foil rolling can be obtained from both of the powder and the cast metal, high vacuum-tight thin foils provide a preform cast beryllium high purity. To reduce oxidation, and more smooth surface it is recommended to roll the foil at temperatures below 430°C with a total reduction of less than 50% and subsequent annealing in vacuum 10…6 mm Hg. st. Reduction per single pass due to rapid hardening beryllium usually no more than 5…10%. The purer the base metal, the lower the rolling temperature may be, and the greater total reduction without intermediate annealing.

The thickness of the foils, received by rolling abroad, typically 30…150 microns. Getting thinner foils get with serious difficulties, although there are reports of possible procedure after rolling beryllium make it 7.5 microns [5].

For high quality vacuum sealed thin foils of beryllium final stage of treatment is recommended to carry out without covers on heated rollers [6].

The published data on the mechanical properties of beryllium foils are very limited.

Table shows the mechanical properties of beryllium foil thickness of less than 100 microns depending on the conditions of their production and grade of the parent metal. The mechanical properties of the foils 100 microns below generally given in the literature for the bulk material of the same grade. The reason for the observed reduction of the strength and plastic properties of thin foils are not fully established.

The mechanical properties of beryllium foil according to [6]

Grade of foil	Manufacturing conditions	$\sigma_{0,2}$, MPa	σ_B, MPa	δ,%
RF1	Cast, transversely rolled at 765°C to a thickness of 100 mkm and chemically thinned to 80 mkm. Annealing 865°C, 12 h	12.8… 17.0	13.9… 19.6	0.2… 2.1
RF2	Cast, transversely rolled at 765 °C to a thickness of 180 mkm and chemically thinned to 100 mkm. Annealing 865°C, 12 h	15.7	25.8	1.2… 2.1
KB1	Cast, hot rolled to a thickness of 380 mkm, grinded to a thickness of 180 mkm and chemically thinned to a thickness of 100 mkm	22.4	17.8… 32.0	0.7… 1.7
BB1	Sintered hot pressed block N-50A, cross rolled to a thickness of 500 mkm, refinished to a thickness of 250 mkm and chemically thinned to a thickness of 125 mkm	23.6	23.5… 31.4	0.3… 0.6

Beryllium foil made from sintered material have higher mechanical properties compared to cast metal foils, but get them vacuum sealed at thicknesses of less than 100 microns is practically impossible. This is due to the presence of particulate material in the second phase breaking denseness of foil.

The aim of the present work was a further optimization of operation thermoplastic treatment of thin sheet of beryllium to produce thin (30…40 microns) beryllium foil, and a study of their structure and properties.

In this work as a raw material for the production of beryllium foils used rolled sheets of 0.8 mm thick beryllium different purity. The beryllium content in the sheets was 99.9, 99.92 and 99.99% (by weight).

Rolling is carried out at temperatures of beryllium 400…750°C with different degrees of reduction in covers made of different materials and without covers in a specially designed rolling mill with heated rollers to 500°C (Fig. 1).

For structural studies of rolled beryllium foils performed X-ray and electron microscopic analyzes. The mechanical properties of the foils were tested for tensile and bending tests using a test design KIPT micromachines. The experimental results can be divided into 3 phases:

1. Choice of processing methods producing vacuum sealed beryllium foils.

2. Investigation of rolling conditions (temperature, degree and direction of deformation) on the structural and mechanical properties of thin foils of beryllium.

3. Influence of purity of the source material on the properties of the foils.

RESULTS OF EXPERIMENTAL STUDIES

In earlier studies carried out by us for rolling sheets of beryllium [3], have been found optimal conditions of deformation in terms of achieving maximum strength and plastic properties. Optimal temperature range corresponds to the temperature 400…700°C and the thermal treatment according to the degree of deformation and the purity of the metal is in the temperature range 650…700°C.

Rolled thin foils and sheets are the closest object on the mechanics of plastic forming, and therefore the founded optimal modes for sheets can be with a sufficient degree of approximation moved to thin foils. In connection with the above, rolling beryllium foils performed in optimum previously found for the sheets.

The materials used in the rolling covers low-carbon and medium carbon steel, stainless steel and nickel. Best results were obtained when using the covers of medium carbon steel type 20X for rolling temperatures 500…600°C. Covers of stainless steel, many researchers believe that it is the best material for this purpose, in the rolling temperatures 400…500°C have strong hardening and for further deformation required the high specific pressure or change of membranes.

For all materials used as covers shells maximum permissible rolling temperature is 600°C. At higher temperature is adhesion of beryllium with the material and separates the foil cover from the foil is very difficult. Reducing rolling temperature to 400°C improves the surface quality of a beryllium foil, but after heat treatment of foil plastic are less than the rolled at 600°C.

Rolling in covers can get vacuum sealed beryllium foil to a thickness of 70…80 microns. On thinner foils there are cracks and gaps formed as a result of the uneven flow of beryllium and cover material. The surface of the foil produced by rolling in a cover, has like a stream pattern, the more pronounced the stronger the material used by the shell. Rolling foils at room temperature and 200°C is accompanied by destruction.

In order to prevent these undesirable effects, has been designed and manufactured vacuum mill with heated to 500°C rollers (Fig. 1). Foils rolled in a vacuum 10^{-6} mmHg with a reduction per pass of 5…10%, and after every 30…50% total deformation carried out relief annealing the internal stress. This method allows receiving schematic foil thickness of 10 microns. In this work studied in detail the structural characteristics of the texture and mechanical properties of the beryllium foils.

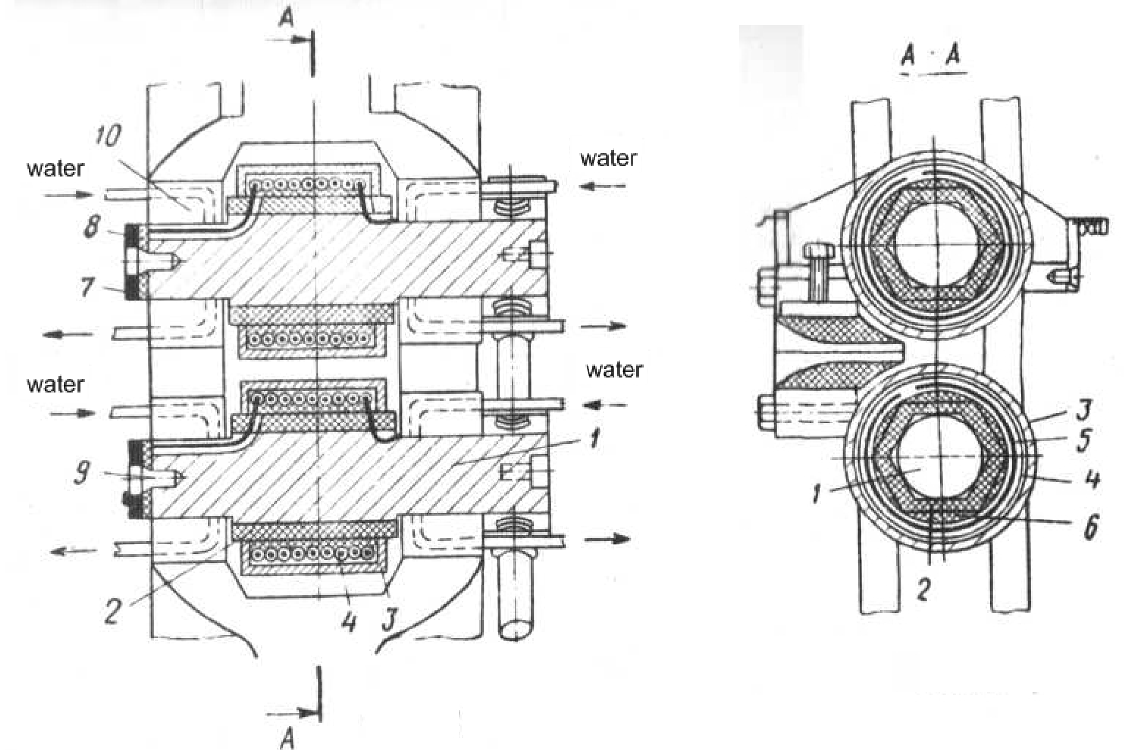

Fig. 1. The design of the rolling mill rolls

STRUCTURAL AND MECHANICAL CHARACTERISTICS OF FOILS

Earlier studies rolled sheets of beryllium showed that increasing temperature of the deformation treatment leads to a weakening the basal texture (0001) [1120], which enhances their isotropic mechanical properties. The high temperature deformation is more favorable for the formation of disoriented dislocation cell structure - one of the most highly effective ways to improve the strength properties of beryllium.

Mechanical properties in the plane of rolled sheet increase with increasing total reduction while increasing the basal texture, whose intensity at a reduction 90% up to 7R. The mechanical properties of sheets in a direction perpendicular to the plane of the sheet, on the contrary, with increased basal texture reduced. Optimum mechanical properties corresponding to their maximum isotropic (in three space dimensions) is achieved at an intensity of basal texture 3-4R.

The studies of the structural state of rolled beryllium foils, depending on their thickness have shown that changes in the intensity of basic poles is nonmonotonic with a maximum (Fig. 2).

The foil with thickness from 800 to 250 microns is continuously increased the basal texture intensity of which reaches a value of 12 R, and then is scattered texture and foil thickness of 20…40 microns texture intensity value R is reduced to a value of 3-4. This nature of reducing the basic texture in the rolling beryllium is observed for the first time.

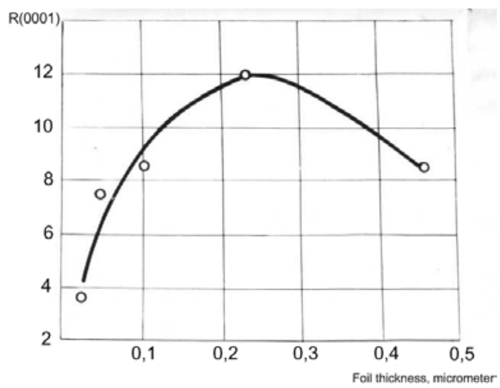

Fig. 2. Intensity of poles (0001) for different thicknesses of rolled beryllium foil

The study of dislocation substructure rolled beryllium foils in original condition and after various heat treatments [7] showed that as the foil thickness from 400 to 20 micrometers (corresponding to the degree of deformation varies from 50 to 97%) occurs the continuous improvement of the dislocation cell structure (Fig. 3).

The cell size is decreased from 1 to 5 microns. In the foils of a thickness of 20 microns in the initial state after rolling cell borders is low dislocation (5.108 cm^2). Inside the cell density of dislocations is also low (≈ 1.109 cm^{-2}) (see Fig. 3,a). In the 200…400 microns thickness foils cell borders are wide, inside the cells a high dislocation density (10^{10} cm^{-2}). The narrowing of the cell borders and reducing the dislocation density is only in the process of deformation after annealing (see Fig. 3,c).

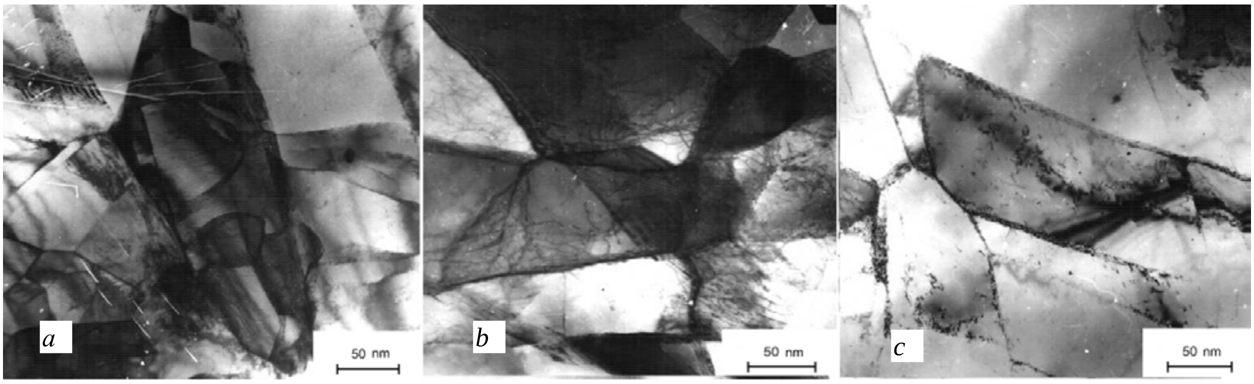

Fig. 3. Electron microscopic structure of thin foils of beryllium:
a – thickness 20 microns; b – 200 microns – initial state; c – thickness of 20 microns – annealed condition

An interesting feature of the substructure rolled foils is the fact that the foil with thickness> 250 micrometers is observed only basic orientation of dislocation cells ((0001) plane parallel to the plane of the foil cells), whereas in foils 100 microns cell basic orientation does not occur at all. The cells have an orientation type (112X). Change the orientation of dislocation cells, presumably due to the feature of the stress state of the metal in thin (less than 250 microns) of beryllium foils.

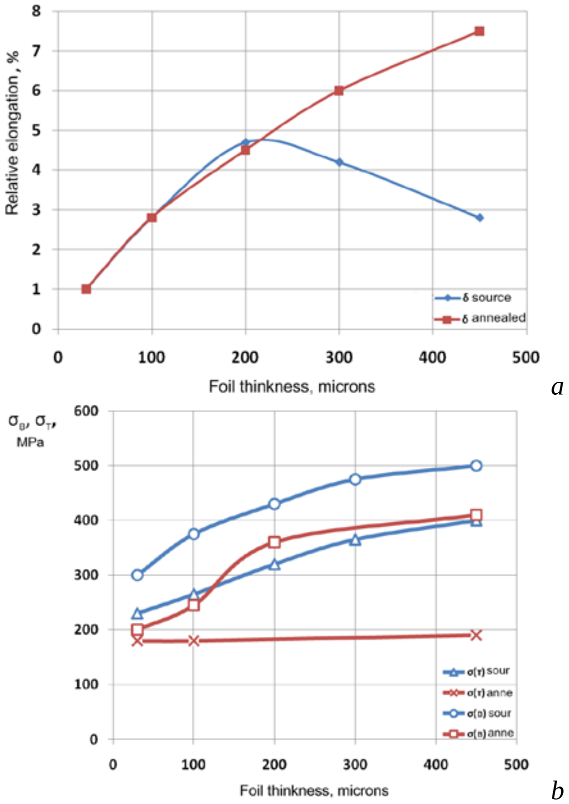

Fig. 4. Influence of thickness on the strength characteristics of beryllium foil in the initial and annealed state (a). Effect of thickness on the plastic properties of beryllium foil in the initial and annealed condition (b)

These structural features of rolled beryllium foils affect on their mechanical properties (Figs. 4,a,b) with decreasing thickness of the foil to 250 microns strength and plastic properties of the foil by the tensile test increases, and then reduced. In this case, the plasticity of foil in bending should be increased, but the change in the properties of the foil in bending is difficult to verify because the foil is very flexible and allow multiple bend on radius of 1 mm. Despite a slight decrease in mechanical properties of beryllium foils with thicknesses of less than 200 microns, the absolute level of tensile properties (σv = 380…420 MPa, $\sigma_{0,2}$ = 250…300 MPa, δ = 2…7%) remains high and much higher than the corresponding characteristics foils, produced by U.S. firms (Table).

These mechanical properties beryllium foil are their tensile testing along the rolling direction. In the transverse direction mechanical properties of the foil is 30…40% lower. Isotropy of the mechanical properties of beryllium foil in different directions foil plane is achieved by periodically changing the direction of rolling of 90° (the so-called cross-rolling).

Noticeable effect on the mechanical properties of the foils has purity of the source beryllium. With increasing purity metal can achieve higher strength and plastic properties. But a decisive influence the beryllium purity has on vacuum density of foils. Foils having a vacuum density 100% can only be produced from high purity beryllium (>99.95%). Testing vacuum density foils, rolled to a thickness of 30…40 microns beryllium purity (99.9%) showed that these foils at 10 cm^2 area is 1-2 points of leakage, and the foil of the same thickness rolled from less pure beryllium (99.8%) on an area of 10 cm^2 are 10 points of leakage, i.e. such foils are practically not vacuum tight.

CONCLUSIONS

1. In KIPT first produced vacuum-tight beryllium foils thickness of 10…50 microns, and studied their structural and mechanical properties. A pilot batch of foils thickness of 20…30 microns for input windows of X-ray detection units.

2. Beryllium rolled foils thickness of 30…100 microns have the following mechanical properties when tested for strength: σ_v = 380…420 MPa, $\sigma_{0,2}$ = 250…300 MPa, δ = 2…7%. Such foils withstand multiple inflection to radius 1 mm. These properties significantly higher than those of similar foils manufactured by USA.

3. For vacuum-tight foil thickness of 20…40 microns is necessary to use beryllium purity not less than 99.95% (by weight).

4. Rolling beryllium in covers of medium carbon steel is possible to produce foils to a thickness of 70…80 microns. Thinner foils (10…50 microns) can be rolled without covers on heated (400…600°C) rolls.

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БЕРИЛЛИЕВЫЕ ФОЛЬГИ ДЛЯ ОКОН СЧЕТЧИКОВ ЯДЕРНОГО ИЗЛУЧЕНИЯ

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На основе оптимизации основных структурных характеристик (зёренной структуры, текстуры, дислокационной субструктуры) определены режимы деформационной и термической обработки фольг бериллия (чистотой > 99,95%), обеспечивающие их высокие механические свойства и оптимизированные режимы их деформации и термообработки. Проанализированы различные технологические приёмы прокатки фольг с целью их рационального применения для практической реализации полученных результатов исследования. Показано, что прочностные и пластические свойства полученных фольг бериллия выше, чем у аналогичных фольг зарубежного производства.

БЕРИЛІЄВІ ФОЛЬГИ ДЛЯ ВІКОН ЛІЧИЛЬНИКІВ ЯДЕРНОГО ВИПРОМІНЮВАННЯ

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На основі оптимізації основних структурних характеристик (зеренної структури, текстури, дислокаційної субструктури) визначено режими деформаційної та термічної обробки фольги берилію (чистотою > 99,95%), що забезпечують їх високі механічні властивості і оптимізовані режими їх деформації і термообробки. Проаналізовано різні технологічні прийоми прокатки фольги з метою їх раціонального застосування для практичної реалізації отриманих результатів дослідження. Показано, що міцність і пластичні властивості отриманої фольги берилію вищі, ніж аналогічної фольги закордонного виробництва.